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Study of Erbium-Doped Semiconductor Devices for Optoelectronics
AASERT Annual Report: July 1, 1993 - June 30, 1994

AFOSR Award F49620-92-J-0323

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Abstract: This project involves the physics of luminescence of erbium (Er) in silicon (Si), with the goal of an electrically pumped Si laser operating at 1550 nm. The AASERT grant funds one graduate student, Mr. Bruce Willner. A liquid nitrogen cooled luminescence measurement system, capable of measuring waveguides, is described. Spectral measurements performed using this system are presented for Er doped glass, as well as two laser diodes at ≈1490nm. Various Si samples were studied. One set were implanted with Er ions by Dr. F. Namavar at Spire Corp. while the other set were grown epitaxially with in situ Er doping using CVD by Prof. Varhue at the University of Vermont. The particular sample preparation conditions are described. Photoluminescence of these samples was studied using the 1490nm, 40mW laser diodes, and an argon laser at 514 nm. No PL signal has been detected from our Er-doped Si samples, although we obtained a spectrum from Er-doped glass. Cathodoluminescence using a 15kV, 20μA current beam, at 10K did not reveal any Er luminescence either. Future plans are described.

1. Introduction

This project consists of electroluminescence (EL) and photoluminescence (PL) studies of erbium (Er) in crystalline silicon (Si). The goal is to develop an electrically pumped Si laser or amplifier operating at 1.54μm for compact and lightweight optical fiber communication systems, which may be integrated with standard Si electronic circuits. We are attempting to directly impact excite the Er in the Si by hot electron bombardment to generate the Er luminescence. By pumping the Er directly using CL, as well as indirectly with PL (i.e. with intermediate electron-hole pairs in the Si), we hope to gain a greater understanding of the excitation and relaxation mechanisms of this system.

2. Measurement Systems

2.1 Photoluminescence System

A liquid nitrogen cooled photoluminescence (PL) system (Figure 1) was designed and constructed to measure luminescence. The system is designed so a sample may be excited by a laser source or by electrical means. The system includes a Stanford Research lock-in amplifier, a thermoelectrically cooled (TEC) EG&G germanium (Ge) detector for infra-red measurements, an IBM-compatible computer, and computer controlled Instruments SA grating monochromator, model HR 320. Additional detectors are available for different wavelength regimes.

The light emitted by the sample travels through a chopper before it reaches the monochromator. After the monochromator the light is focused on the detector. The TEC Ge detector is reverse biased with 4 volts in series with a load resistor (typically 100k to 500k Ohms). The voltage across the load resistor is measured by the lock-in amplifier which filters out all signals which are not frequency matched with the chopper. The computer controls the lock-in and monochromator to scan over a range of wavelengths.

The PL system is designed to allow for the measurement of bulk samples as well as waveguiding samples. To measure a bulk material, such as a piece of Er-doped glass, the excitation beam is collimated and directed through the sample. Following the sample a lens gathers a portion of the output light. For waveguiding samples, the collimated pump beam is focused on the edge of the waveguide with a microscope objective and the output light is gathered by another objective. The output light is then focused on the slit of the monochromator by a cylindrical lens.

Er-doped Si luminesces quite weakly at room temperature. Therefore, a cryogenic chamber is used to hold the sample. Windows on the chamber allow light to be coupled into and out of the sample. The chamber is evacuated by a roughing pump and is then cooled with liquid nitrogen.

We presently have two 40mW fiber coupled laser diodes for pumping. The output of these two laser diodes are centered at 1485 and 1495nm as seen in Figure 2. An interference filter blocks the spontaneous emission noise of the pump sources which extend into the Er luminescence band. One problem discovered with the system, however, was that the monochromator allowed leakage of the 1.48-1.49μm wavelength laser to pass through near the 1.54μm range of the anticipated luminescence. The monochromator reduces this light by approximately 50dB, but it is still detected by our lock-in measurement system. This problem was corrected by adding a long wave pass optical filter at the output of the monochromator, which blocks light with wavelengths less than ~1.52μm. We will also be receiving a 100mW 980nm laser diode from IBM in the near future.

Once the system was completed, the PL of a block of Er-doped glass, provided by Dr. Kevin Malone of NIST, was measured. This confirmed that the measurement apparatus operated correctly. (Figure 4)

2.2 Cathodoluminescence System (Figure 3)

There is also a helium cooled cryogenic CL system that can reach temperatures of 6 Kelvin. The electron gun can provide currents up to 25μA and an accelerating potential of 20kV. The luminescence is measured with a Perkin Elmer automated prism spectrometer and either the Ge

detector mentioned above, or a dry ice cooled PbS detector for infra-red measurements. The output is connected to a Princeton Applied Research lock-in amplifier. This measurement system is also computer controlled.

3. The Samples

There were two groups of Er-doped Si samples available for measurements during the past year. One group is epitaxially grown Er-doped Si on a <100> Si substrate. These samples were grown by Prof. W. Varhue at the University of Vermont. The second group is Er-implanted SIMOX supplied by Dr. F. Namavar at Spire corporation. In addition, we have a number of Er implanted Si on sapphire samples.

3.1 Epitaxial Er-doped Silicon

Most Er-doped Si is simply crystalline Si which is implanted via an ion beam. Dr. Varhue is using the chemical vapor deposition technique to epitaxially grow a layer of Er-doped Si on top of a silicon substrate. The epitaxial Er-doped wafers are doped with approximately 10^{19} cm^{-3} Er. Some of the samples are co-doped with oxygen at concentrations approximately ten times that of the Er. The substrate is oriented in the <100> plane. These samples were grown at low temperatures (350 C). The samples were not annealed. Light confinement in the Er:Si layer is an area to be studied, and is essential for waveguiding devices.

3.2 SIMOX

The SIMOX samples provided by Dr. F. Namavar were implanted with Er^{2+} ions at 400keV. These samples are also codoped with oxygen at various densities. A SIMOX wafer is a silicon wafer with a crystalline surface layer above a narrow layer of SiO_x. The SIMOX samples are advantageous for the fabrication of waveguides. An oxide layer beneath the Er-doped Si layer provides optical confinement to any light in the Er-doped Si layer, which has a much larger index. Otherwise, much of the light would travel in the optically inactive portions of the wafer.

3.3 Erbium Doped Si On Sapphire

Planar waveguides in Erbium implanted Si grown on sapphire (Al_2O_3) substrates have been investigated. Although there is a lattice mismatch between Si and sapphire, the Si films are crystalline and of good quality. The refractive indices of Si and sapphire are 3.4 and 1.8 respectively. The large index difference in the two materials makes the material system a good candidate for fabricating single mode waveguides with strong confinement of the mode. In the active medium, a thin layer of Er-doped Si, the Er ions will then have a greater overlap with the pump field. Unfortunately, the mode matching to optical fibers is poor.

3.3.1 Implantation

Six 2-inch wafers with Si films (various thicknesses) grown on sapphire (Al_2O_3) were donated by Dr. Richard Soref at the USAF Rome Laboratory. One whole wafer, and one half of another wafer were implanted with Erbium by Dr. Stephen Withrow at Oak Ridge National Laboratory . The implantation conditions are listed in Table I below:

Table I - Implantation Conditions:

Sample:	Implants:
1. ErSi:	0.69×10^{14} Er^+/cm^2 at 1 MeV and
(1 μm Si on sapphire)	0.31×10^{14} Er^+/cm^2 at 600 keV
2. ErSi:	0.69×10^{14} Er^+/cm^2 at 1 MeV and
(1.5 μm Si on sapphire)	0.31×10^{14} Er^+/cm^2 at 600 keV

The implantation depths were calculated to be 329 nm for 1MeV with a straggle of 23 nm, and for 600keV a depth of 190 nm with a straggle of 16 nm.

3.3.2 Anneal

The samples were rapid thermal annealed (RTA) by Ms. Carol Lee at The Army Research Lab., Ft. Monmouth , NJ, through a collaboration with C. Lee, K.K. Choi, and Wayne Chang. The samples were annealed in a nitrogen atmosphere. The Si flaked off sample 1 during the first anneal, probably due to the mismatch in coefficients of thermal expansion in Si and sapphire. Visual inspection showed that the following anneals of the remaining samples went well. However, the temperature may not have been sufficient to repair all the damage to the lattice that occurred during the implantation. The relevant parameters used in the rapid thermal anneal are listed in Table II below. The sample numbers correspond to the implant conditions listed above in Table I.

Table II - Annealing (RTA) parameters for Si on sapphire:

	Sample:	Anneal	Temp.	Duration	Ramp time
1.	ErSi	RTA	900C	15 sec	?
2a.	ErSi	RTA	600C	15 sec	170 sec
2b.	ErSi	RTA	600C	15 sec	113 sec
2c.	ErSi	RTA	600C	15 sec	74 sec

3.3.3 Waveguide Preparation

Waveguides were fabricated using reactive ion etching (RIE). Standard lithography, metal evaporation and lift-off techniques were used to create a mask for the RIE. The plasma used was a polyetch (CF_4). We etched two different depths: 0.9µm and 0.6µm. After the RIE the remaining metal on top of the waveguides was removed with a wet chemical etch. The samples were then cleaned with Acetone, Methanol and trichloroethylene, prior to examination with a scanning electron microscope (SEM), as shown in Fig. 5.

There were problems polishing the endfaces, which is necessary for good optical coupling. We were not successful using a Buehler Ecomet 3 wafer polisher with various diamond polishing pads. After discussions with technicians and scientists in industry and at NIST (National Institute of Standards and Technology) we decided to abandon the polishing in favor of using a wafer saw. A wafer saw from Assembly Technologies, model 1006, is scheduled to be purchased by another research group in our department. The wafer saw process should be simpler and less time consuming to characterize, since similar material systems have been investigated and controlled by the vendor. The sawing of Si on sapphire will be characterized using an Assembly Technologies metal/resinoid blade with various diamond grit sizes, blade (spindle) speeds (ranging from 15-25 K rpm) and feed rates.

3.4 Discussion

The wafer saw has now arrived, and we are in the process of characterizing the process of cutting optical quality end faces on the Si on sapphire, as well as on epitaxial Si on Si..

4. Optical Measurements

4.1 Absorption In Epitaxially Grown Erbium-doped Silicon

Absorption spectra of three, 3 inch wafers of epitaxially grown Er:Si layers grown on Si substrates were measured. The epitaxial layers were grown by Prof. Varhue at The University of Vermont. The absorption measurement was performed using a Cary spectrophotometer. A weak absorption signal was found at 1.54µm in wafer UVM-ER9 (Figure 6). There was no detectable Er related absorption on the other two samples.

PL and CL were also performed on these samples with no luminescence signal detected.

4.2 Photoluminescence

Measuring the luminescence of Er-doped Si samples turned out to be more difficult than measuring the Er-doped glass. The Si samples were mounted inside the cryogenic chamber. The first measurements involved shining the laser straight through the sample. The beam travels through the Er-doped surface of the sample as well as the substrate. The Si bandgap of 1.1 eV provides a substrate which is optically transparent for the relevant wavelengths. The difficulty with this sort of measurement is the very thin Er-doped region which only absorbs approximately 5 $* 10^{-6}$ of the incident pump light, leading to very small signal powers. A theoretical luminescence calculation for the sample shows that luminescence emission will be very weak. The theoretical result [1] for our system is only 16μV of signal for 1W of 1480 nm pump power.

4.3 Luminescence Measurement At NIST

In cooperation with NIST, we excited the Er indirectly by optically generating electron-hole pairs in the Si. The Er atoms can be excited as the electron-hole pairs recombined. This is how other groups have found Er luminescence in Si and other materials.

Dr. K. Malone at the National Institute of Standards and Technology provided access to high powered Ti-sapphire and argon lasers for our use to measure some GaN and Si samples. The Ti-sapphire laser emitted up to 1 Watt of 980nm light and the argon laser could produce nearly 10 Watts of light at 514nm.

Time limitations allowed us to measure only one silicon sample (pumping down and cooling the cryogenic chamber takes a significant amount of time). This sample was excited with the Ti-sapphire laser, but no Er luminescence was detected.

4.4 Cathodoluminescence Measurement

Several SIMOX and epitaxially grown samples were studied in the cryogenic CL system. The CL system was cooled to approximately 6 K. Electrons were fired at the sample with a current of 20μA and 15kV potential. A luminescent Er-doped GaN sample was used as a standard for alignment. No luminescence was found in any of the Er:Si samples.

A problem with the CL measurements was the high noise level in the aging PbS detector. In the future we expect to acquire a North Coast liquid nitrogen cooled Ge detector.

4.5 Annealing

We theorized that the reason for the optical inactivity of the Er was a need for interaction between the Er and oxygen atoms in order to place the Er in the optically active Er^{3+} state. The epitaxially grown samples were never heated above 350 C, so the atoms were not free to move in the Si lattice and will not interact. The implanted SIMOX samples were only annealed with RTA which does not allow adequate time for the atoms to move and interact. In order to allow the oxygen time to move in the lattice, and possibly bond with Er, we annealed some of each sample type for 5 hours at 625 C followed by 20 minutes at 900 C. The anneal was performed in a furnace with a mostly nitrogen atmosphere. Following the anneal, CL measurements were performed. At this time, these measurements have yet to yield any Er luminescence signals. However, we are still studying these samples.

4.6 Waveguiding

We are beginning to employ waveguiding in order to enhance any PL signal from the Er. Coldfingers for the PL system were designed to hold samples with edges facing the windows. Objective lenses are used to focus the pump beam into the guide and to gather the light leaving the guide. A Micronviewer 7290 Infrared Camera was acquired in order to view the infrared laser beam and assist in aligning the pump beam into the guiding structure. The first waveguide samples measured were slab waveguides of Er-doped Si on Si grown by Prof. Varhue.

The alignment is difficult due to the small height of the Er-doped Si layer (approximately 1μm). Also, as a result of coupling light into the substrate as well as the Er doped region, the light exits the wafer throughout the Er-doped and undoped regions of the endface. This makes it difficult to ascertain whether the pump beam is incident upon the Er-doped layer or if it is incident upon some other part of the wafer. We expect to overcome these difficulties in the future. We had little difficulty aligning the pump beam to a thin film polymer on glass, which was produced by Prof. A. Mickelson at the University of Colorado.

5. Other Work

Wang and Wessels at Northwestern University have measured thermal quenching of Er in GaP[2]. They theorize that the luminescence falls with increased temperature because of Er-related isoelectronic trap energies. The paper proposes that bound excitons are an intermediary in the excitation of the Er centers. As temperature increases, the bound excitons are more likely to break up. This offers an explanation for the poor luminescence of Er:Si at room temperature. Co-doping the samples to form Er compounds may increase the luminescence in Si by increasing the depth of the trapping energy level, resulting in stronger binding of the excitons.

At the Electronic Materials Conference held June 22-24, 1994 at the University of Colorado at Boulder, J. Michel of MIT presented a paper entitled "Er-O and Er-F Reactions in Silicon"[3]. A model for optimal dopant concentrations was developed and combined with experimental results. The group concentrated on Fluorine co-doping, which was found to produce 100 times greater emission than oxygen co-doping. They also used significantly lower doping concentrations than we have. The crux of the paper was the use of annealing to optimize emission. They found that annealing a sample with $5x10^{17}$ cm^{-3} Er and $1x10^{18}$ cm^{-3} O or F at 900 C for 0.5 hours optimized emission. The optimal temperature decreased to 800 C for F concentrations of $4x10^{18}$ cm^{-3}. The process model suggested that the Er emission was optimized when the concentration of ErF_3 was greatest.

6. Future Work

6.1 Improved Detectors

It has come to our attention that most other groups studying Er-doped Si employ more powerful lasers and very highly sensitive photodetectors (generally a North Coast detector which provides an output of $>10^9$ Volts per Watt optical input power). We have taken delivery of a damaged liquid nitrogen-cooled North Coast detector donated by the National Renewable Energy Laboratory in Golden, Colorado. We are repairing the detector on our own and the results look promising. In addition, we are designing and building a low-noise amplifier for the thermo-electrically cooled Ge detector.

6.2 Waveguides

Shortly we will be fabricating Er-doped Si rib waveguides from the various samples. These waveguides will enhance any Er PL signal present. A modeling program has been written which calculates mode wavenumbers and the shape of the propagating field in a rib waveguide. This software is based upon Chan Beta software which was developed at the University of Colorado by Marc Surette and Prof. Alan Mickelson. The software employs the effective index approximation to calculate the modes.

6.3 Fabrication

We plan to employ several techniques for fabricating the Si waveguiding structures in order to compare the quality of the resulting structures. These techniques will include various wet chemical etches as well as RIE. Our first guides will employ a KOH wet chemical etch.

To etch a pattern with KOH a mask is needed. The standard mask for KOH use on silicon is simply SiO_2 grown on the wafer. This is unacceptable for our purposes because any growth of SiO_2 will consume some of the Er-doped surface layer of the samples. Instead, we will use gold

as a mask.

Photoresist will be put on the sample which will then be patterned according to a waveguide mask. The photoresist will then be developed, leaving the desired waveguide areas bare. A few nanometers of gold will be evaporated onto the patterned sample. Following the evaporation, liftoff will remove the photoresist and any gold layered on top of the photoresist. This will result in a sample with gold lines defining the waveguides. The Si will then be etched with KOH to form waveguides. Finally, a gold etch will remove the remaining gold. The dimensions of the waveguides will be chosen according to the results of the waveguide modeling program. The facilities for the etch process are being provided by Prof. Alan Mickelson.

6.4 Contacts and Electroluminescence

We also plan to make contacts on some of the samples and electrically pump them. Electrically pumping the sample may be more efficient than optical pumping.

6.5 Index Analysis

It would be beneficial for the design of waveguides if we knew the precise index of refraction of Er-doped Si for various concentrations of Er. For that reason we plan to design an ellipsometric system to measure the index of Er-doped Si films at 1.54μm.

7. References

[1] J.I. Pankove, R.J. Feuerstein, Annual Report: Study of Erbium-Doped Semiconductor Devices for Optoelectronics, August 1, 1992 - June 30, 1993, Air Force Award F49620-92-J-0323.

[2] X.Z. Wang, B.W. Wessels,"Thermal quenching properties of Er-doped GaP" *Applied Physics Letters* Vol 64(12) pp. 1537 (1994).

[3] F.Y.G. Ren, S. Dunham, J. Michel, B. Zheng, L. Giovane, L.C. Kimerling, "Er-O and Er-F Reactions in Silicon," to be published in Proceedings of Electronic Materials Conference, Boulder, CO (1994).

8. LIST OF FIGURES

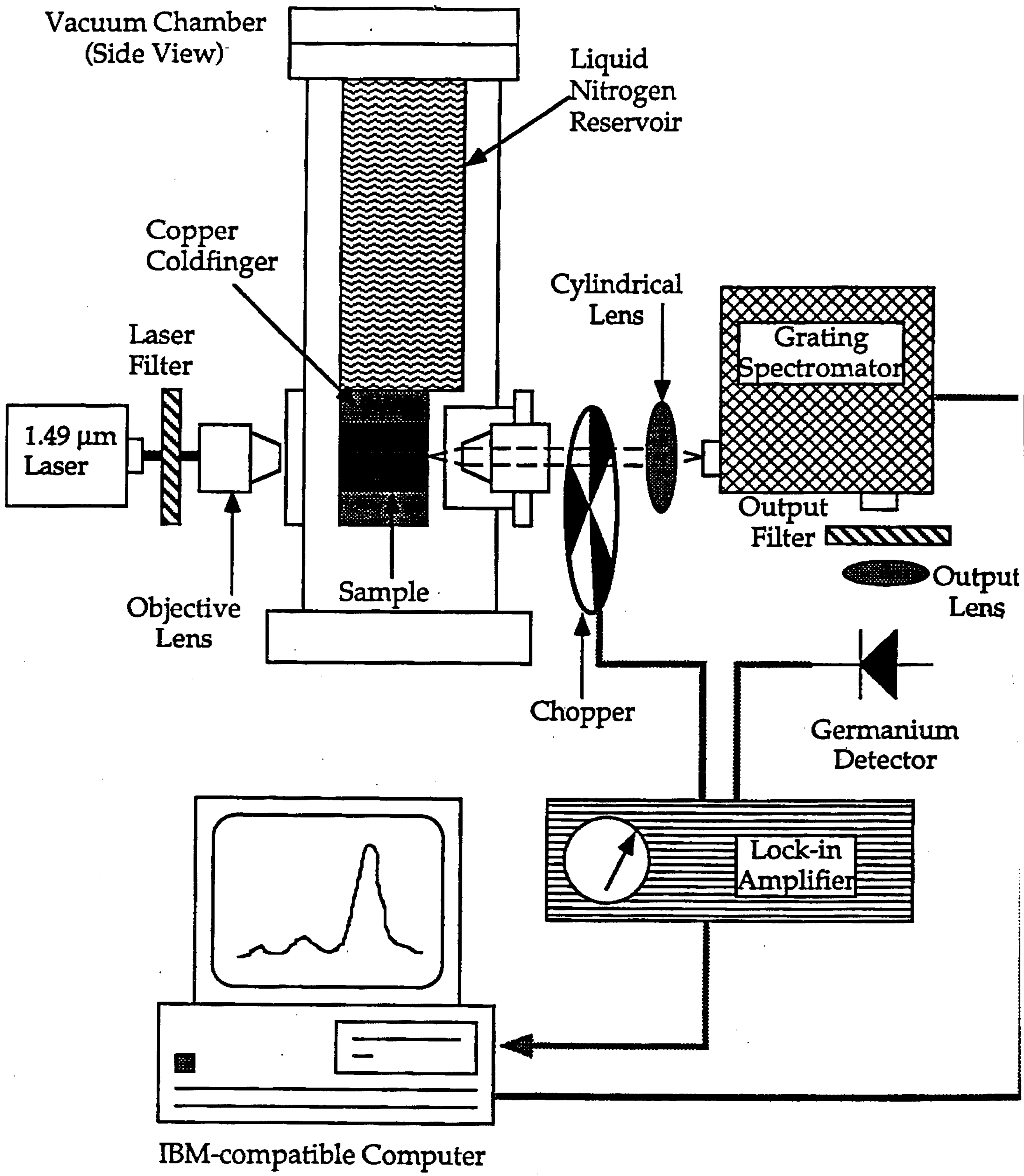
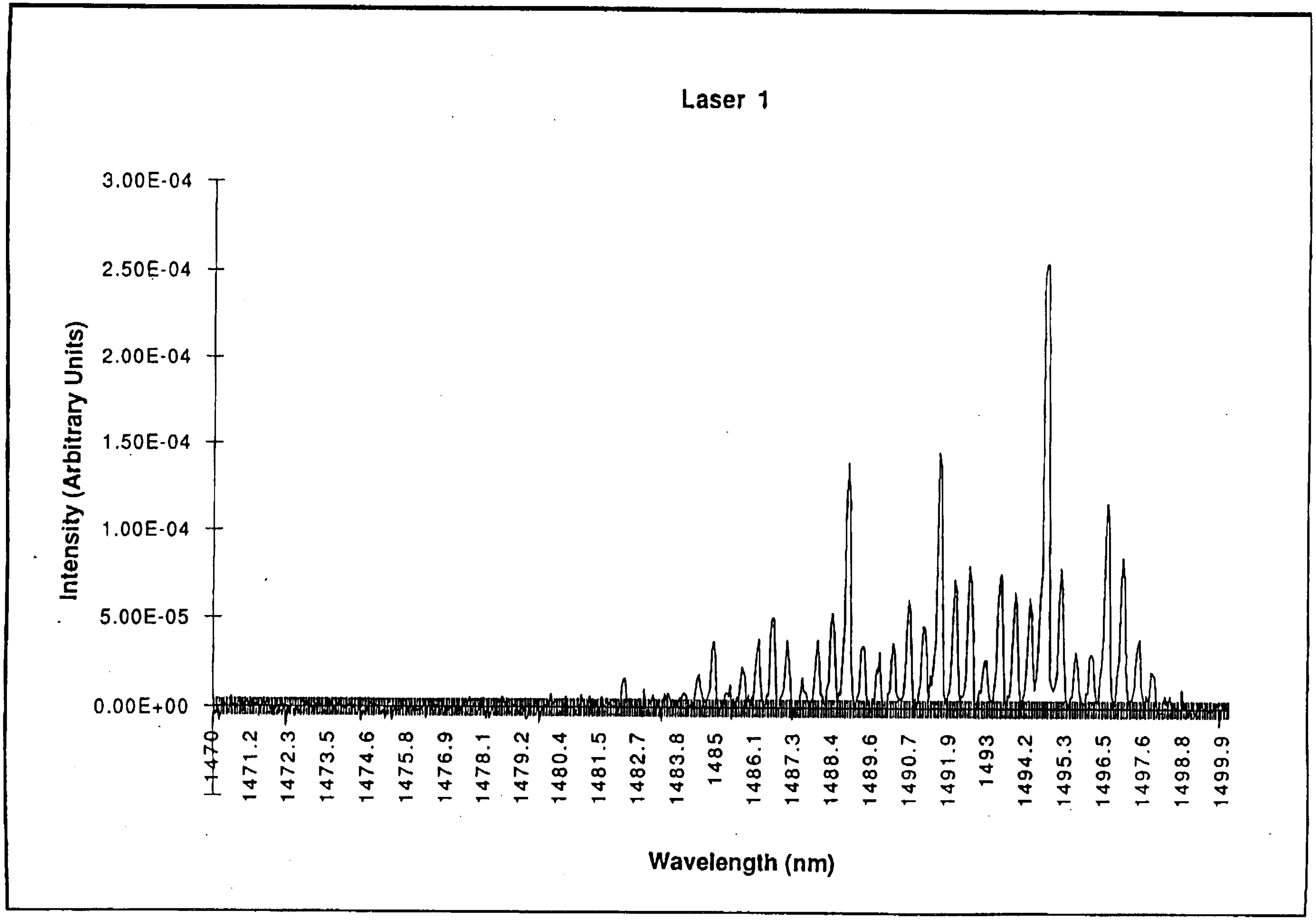


Figure 1: Computer controlled cryogenic waveguide measurement system.



<u>Figure 2a: Spectrum of Laser 1.</u>
(Laser Current = 180mA, Slit widths = 50μm and 70μm)

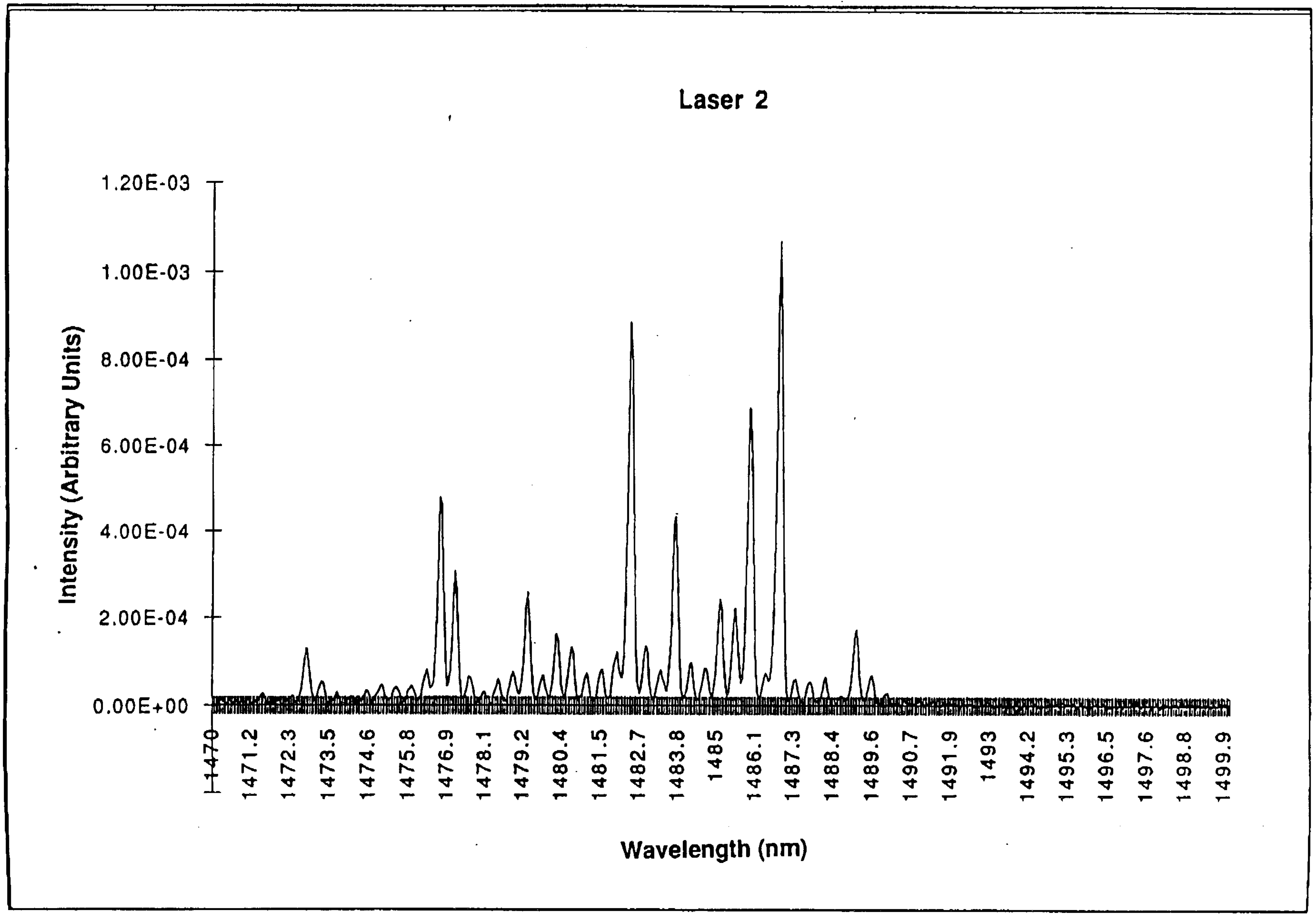


Figure 2b: Spectrum of Laser 2.
(Laser Current = 170mA, Slit widths = 50μm and 70μm)

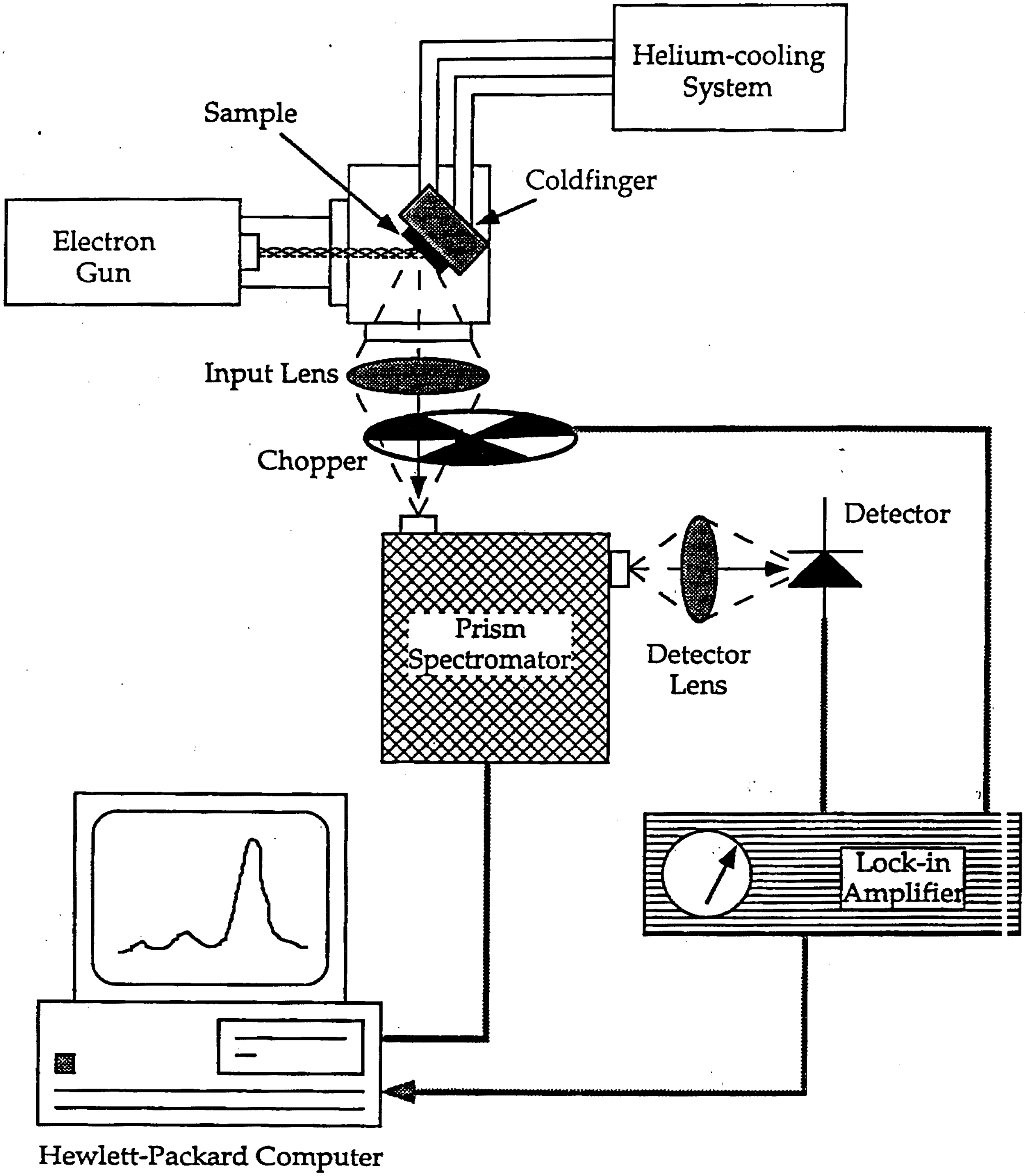


Figure 3: Computer controlled cryogenic cathodoluminescence system.

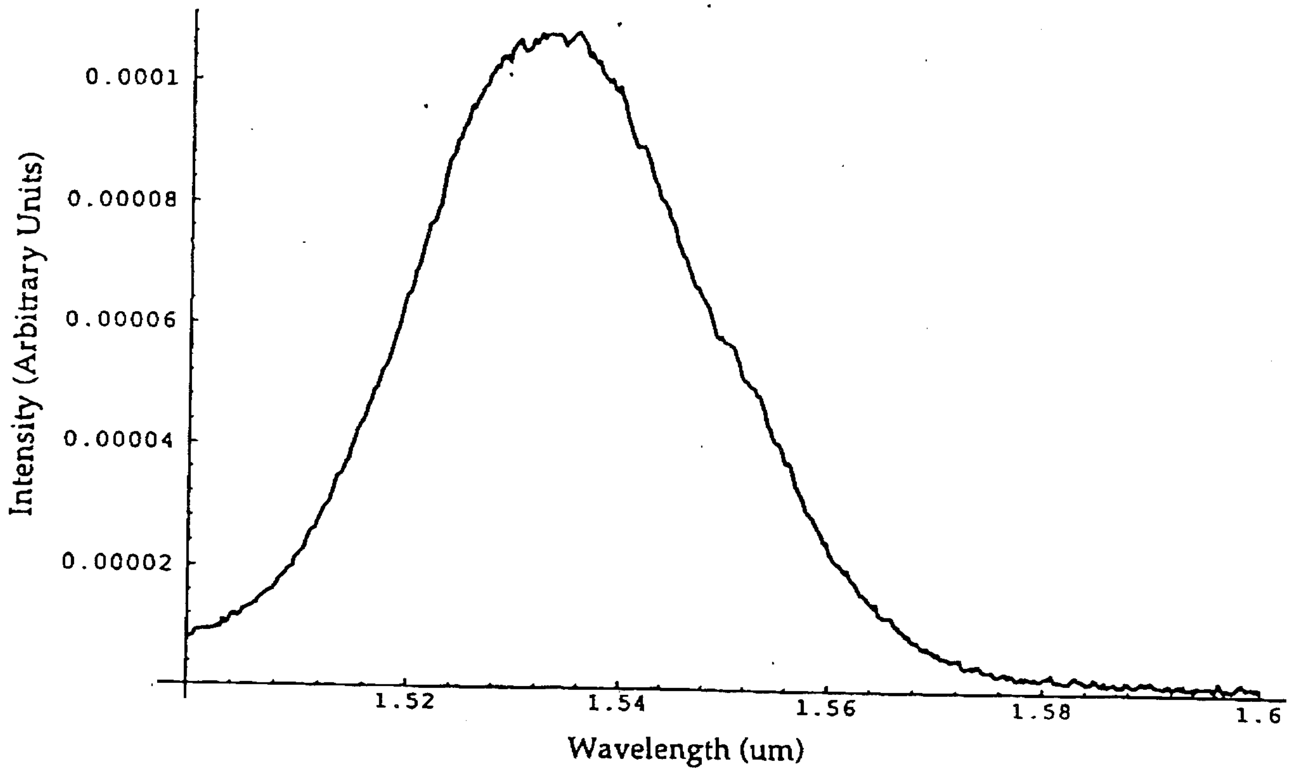
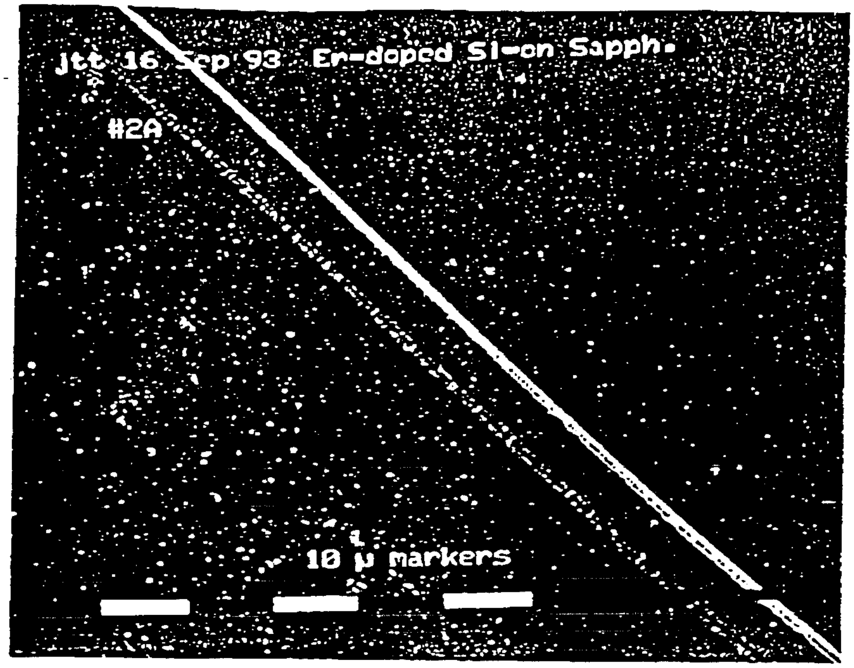


Figure 4: Photoluminescence of Er-doped Glass.
(Pumped with 1490 nm diode laser 1,
Slit widths = 1mm and 25μm)



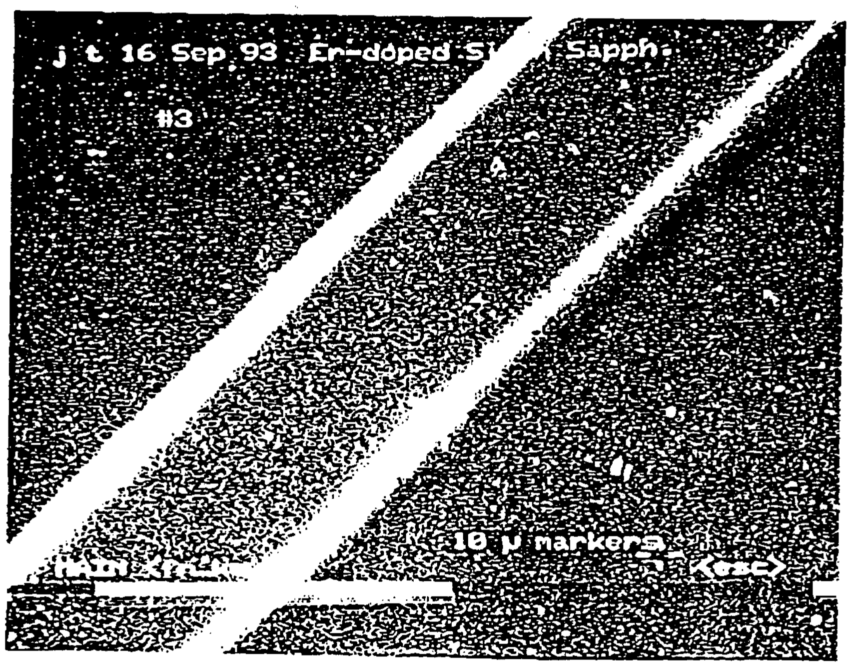


Figure 5: SEM Image of Er-doped Si on Sapphire Waveguides.

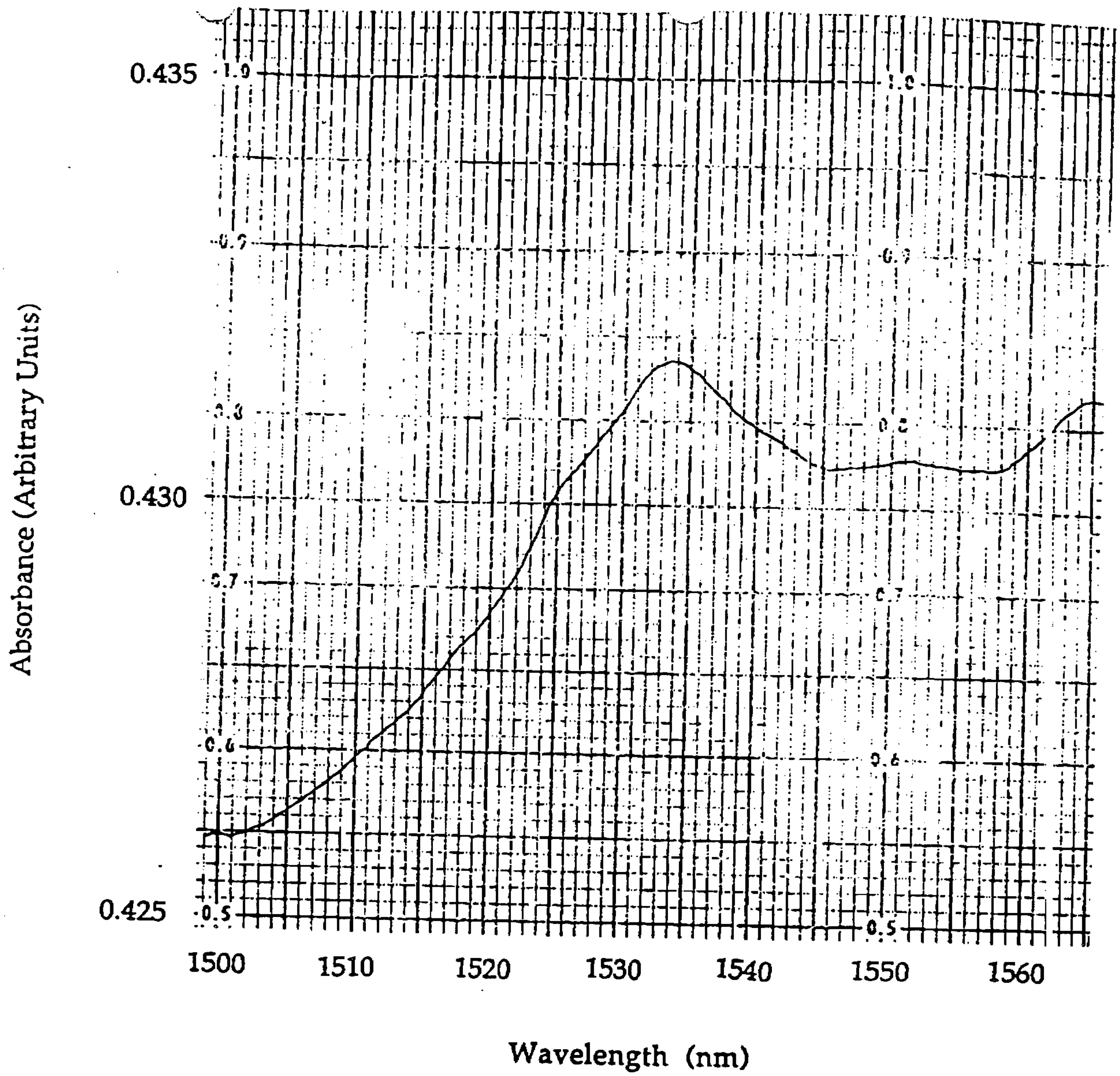


Figure 6: Absorption of Epitaxially Grown Er-doped Si.
(Sample Er9)